said improvement comprising continuously monitoring the aspect ratio of said acicular calcium sulfate alpha hemihydrate crystals and adjusting the amount of alum used to form said slurry, the amount of alum being sufficient to maintain the aspect ratio of said crystals to at least about 5:1 and no greater than about 50:1.

14. (Previously Amended) The improved process of claim 13, wherein the amount of alum is adjusted to maintain the aspect ratio of said crystals to at least about 10:1 and no greater than about 50:1.

### **REMARKS**

Responsive to the Office Action dated October 19, 2005, applicants have canceled claims 1 and 3 through 4, without prejudice, and amended claim 5, leaving claims 5 through 14 in issue. All of the claims in this case are set forth in "Jepsen" format, wherein the process described in US Patent 5,320,677 to Baig is the acknowledged prior art. The patentable improvement over the prior art is the use of aluminum chloride, chlorine or alum in the process for calcining gypsum to control the aspect ratio of the acicular calcium sulfate alpha hemihydrate crystals being produced. The present invention provides a mechanism to control the aspect ratio of the acicular calcium sulfate alpha hemihydrate crystals being produced even in the presence of variable contaminants in the raw material mix.

As filed, claims 1, 3 and 4 (now cancelled) were directed to the use of various crystal modifiers to reduce the temperature and/or time necessary to form acicular calcium sulfate alpha hemihydrate crystals. As filed, claim 5 defined a Markush group of crystal modifiers that could be used to increase the aspect ratio of the acicular calcium sulfate alpha hemihydrate crystals. Claim 5 (and dependent claims 6 – 8) now specifies the use of either aluminum chloride or chlorine to increase the aspect ratio of the acicular calcium sulfate alpha hemihydrate crystals being produced. Applicants' specification (at page 8, lines 18-20) points out that aluminum chloride and chlorine "act as crystal modifiers to change the crystal morphology to the desired configuration."

Claims 9 and 13 (and dependent claims 10 – 12 and 14) specify the use of alum in the process for calcining gypsum to control the aspect ratio of the acicular calcium sulfate alpha hemihydrate crystals being produced.

Gypsum, calcium sulfate dihydrate, is a crystalline material having two molecules of water for each molecule of calcium sulfate in the crystals. Alpha calcium sulfate hemihydrate is also a crystalline material, but having only one half a molecule of water for each molecule of calcium sulfate in the crystals. As is explained in US Patent 5,320,677 to Baig, when a slurry of gypsum is heated to an elevated temperature, such as 140° C, an average of one and one half molecule of water is driven out of the calcium sulfate dihydrate molecule. More accurately, this process forms a solution of dissolved calcium sulfate that may be

preferentially precipitated to form crystalline acicular calcium sulfate alpha hemihydrate having an average of one half molecule of water for each molecule of calcium sulfate in the crystals. Acicular calcium sulfate alpha hemihydrate has a atomic crystalline lattice and structure that differs from that of the starting material, calcium sulfate dihydrate.

If the temperature of the slurry is raised further and/or the time at elevated temperature is extended, the chemical equilibrium shifts and a calcium sulfate crystal without any water is formed, calcium sulfate anhydrite. Calcium sulfate anhydrite has a third type of crystalline lattice, but having no water within the calcium sulfate crystal. Calcium sulfate anhydrite has chemical and physical properties quite different from those of calcium sulfate alpha hemihydrate or the calcium sulfate dihydrate, the starting material. Calcium sulfate anhydrite is not useable in the process of the present invention.

Calcium sulfate hemihydrate and calcium sulfate anhydrite are different materials, with different crystal forms. The lattice symmetry of alpha hemihydrate and soluble anhydrite are rhombohedral and hexagonal respectively. The lattice spacings (nm) for a, b, and c are 0.683, 0.683 and 1.270 for hemihydrate, and 0.699, 0.699, and 0.634 for soluble anhydrite respectively. Specific gravity of the crystals are 2.757 for alpha hemihydrate and 2.580 for soluble anhydrite. The crystal structure (i.e. lattice symmetry, lattice spacings and specific gravity) of gypsum, calcium sulfate dihydrate, differs from the crystal structure of either

alpha hemihydrate or soluble anhydrite. (see Ullman's Encyclopedia of Industrial Chemistry, Vol. A4, Calcium Sulfate, Table 2, page 558)

All of the claims in issue stand rejected under 35 USC §103 as being obvious based on US Patent 5,320,677 to Baig in view of US Patent 3,835,219 to Jaunarajs et al. Claims 5 -8 require aluminum chloride or chlorine to be included in the water, gypsum and cellulosic fiber slurry that is fed into the autoclave to convert the gypsum into acicular calcium sulfate alpha hemihydrate. All of these claims require the hemihydrate emerging from the autoclave to have an increased aspect ratio.

Claims 9 through 14, also stand rejected under 35 USC §103 as being obvious based on US Patent 5,320,677 to Baig in view of US Patent 3,835,219 to Jaunarajs et al and a 1995 paper by Spiring. Claims 9 through 14 require alum to be included in the water, gypsum and cellulosic fiber slurry that is fed into the autoclave to continuously convert the gypsum into acicular calcium sulfate alpha hemihydrate. Claims 9 through 14 require the aspect ratio of the hemihydrate emerging from the autoclave to be continuously monitored and the aspect ratio of the hemihydrate to be controlled by adjusting the level of the alum going into the autoclave.

## **BAIG**

US Patent 5,320,677 to Baig discloses the process set forth in the preamble portion of all of the applicants' independent claims. The Baig process

includes 4 essential steps that are set forth in the preamble of the independent claims. First, a slurry of water, gypsum and cellulose fiber is heated under pressure to form acicular calcium sulfate hemihydrate crystals. Second, the slurry is dewatered. Next, the dewatered slurry is shaped. Finally, the hemihydrate crystals in the shaped slurry are rehydrated back to gypsum.

As the Examiner pointed out in the Office Action, Baig suggests the use of organic acids as crystal modifiers. At Col. 6, lines 42-45, Baig states only that organic acids can be added to the slurry can "to stimulate or retard crystallization or to lower the calcining temperature." In other words, Baig suggests that crystal modifiers may be used to lower the calcination temperature or speed up the calcination process. Baig does not suggest the organic acids affect the aspect ratio of the hemihydrate crystals. Baig fails to suggest the use of aluminum chloride, chlorine or alum for any purpose.

Baig does not discuss the aspect ratio of the acicular calcium sulfate hemihydrate crystals being produced. Although Baig describes a continuous process for making a composite material, there is no discussion of monitoring any aspect of the morphology of the hemihydrate crystals being produced. Accordingly, Baig provides no suggestion to continuously monitor the aspect ratio of hemihydrate crystals being produced nor does he provide any suggestion of controlling the aspect ratio hemihydrate crystals being produced, as required by applicants' claims 9 - 14.

# JAUNARAJS ET AL

US Patent 3,835,219 to Jaunarajs et al is directed to a method of preparing fibrous soluble calcium sulfate anhydrite that avoids the formation of any calcium sulfate hemihydrate. The Jaunarajs et al method does not contemplate the formation of any type of calcium sulfate fibers in the presence of cellulose fibers, as required by applicants' claims. The Jaunarajs et al patent states the process selectively produces fibrous soluble anhydrite "to the virtual exclusion of non-fibrous anhydrite, insoluble anhydrite, and/or hemihydrate" (Col. 2, lines 18-20).

Jaunarajs et al also describes the use of "crystal habit modifiers" that are "suitable for the formation of the fibrous soluble anhydrite." (See Col.1, lines 66-67 and Col. 3, lines 11-12). Most of the "crystal habit modifiers" disclosed by Jaunarajs et al are organic acids, although Jaunarajs et al lists some inorganic modifiers including aluminum sulfate (the major component of alum). Jaunarajs et al does not mention using aluminum chloride or chlorine as crystal modifiers or for any other purpose. Moreover, Jaunarajs et al does not specify the function of any of the disclosed "crystal habit modifiers." Those skilled in the art would understand Jaunarajs et al to teach that the "crystal habit modifiers" promote the formation of fibrous soluble anhydrite apparently "to the virtual exclusion of non-fibrous anhydrite, insoluble anhydrite, and/or hemihydrate". Like the Baig

disclosure, Jaunarajs et al fails to suggest that these "crystal habit modifiers" have any impact on the aspect ratio of any type of calcium sulfate fibers.

Those skilled in the art would recognized, however, that the fibrous soluble calcium sulfate <u>anhydrite</u> produced by the Jaunarajs et all process would not be suitable for use in the Baig process. Calcium sulfate anhydrite is notoriously difficult to rehydrate. The last step of the Baig process, as set forth in applicants' claims, is to rehydrate the acicular calcium sulfate alpha hemihydrate crystals back to gypsum to bond the cellulose fibers into a composite material. As a practical matter, the rehydration step in applicants' continuous process must take place within a short time. Accordingly, the fibrous calcium sulfate <u>anhydrite</u> product of Jaunarajs et all would be difficult or impossible to use in the rehydration step of the Baig process defined by applicants' claims because the rate of rehydration must be synchronized with other steps of the process to achieve satisfactory final composite properties (e.g. see US Patent No.: 6,197,235 B1, Figure 6, Claim 9, 16, etc.).

The Jaunarajs et al soluble anhydrite fibers are used to reinforce a variety of organic polymeric resins (Col. 4, lines 17 – 20). Those skilled in the art would be well aware that anhydrite fibers don't need to be rehydrated for use in organic polymers. Fibers containing water (such as applicants' hemihydrate or any rehydrated version of these fibers) would not be suitable for use in organic polymers because fibers containing water would release water/steam during

conventional polymer melting and molding operations and thus interfere with the properties and stability of the organic polymers. In other words, Jaunarajs et al teaches the production of fibers that can't be used in applicants' process and applicant prepares fibers that can't be used in place of the fibers described by Jaunarajs et al.

It is submitted that one skilled in the art would not apply the teaching of Jaunarajs et al to the process disclosed by Baig because of the hydration problems with the fibrous calcium sulfate anhydrite product of Jaunarajs et al. It would be apparent to one skilled in the art that the fibrous calcium sulfate anhydrite product of Jaunarajs et al just wouldn't work in the Baig process. If the teaching of Jaunarais et al concerning "crystal modifiers" were combined with Baig, as asserted by the Examiner, and through the use of some form of serendipity one skilled in the art selected aluminum sulfate as the crystal modifier, at best, one skilled in the art might conclude that aluminum sulfate could be used to the speed up the calcination process or to lower temperature of a gypsum calcination process. More likely, one skilled in the art would expect the aluminum sulfate to promote an anhydrite "to the virtual exclusion of non-fibrous anhydrite, insoluble anhydrite, and/or hemihydrate" as taught by Jaunarais et al. Based on the teachings of Baig in the light of Jaunarajs et al, one skilled in the art would have no basis to believe the aspect ratio of the acicular calcium sulfate hemihydrate crystals would be altered in any manner.

Jaunarajs et al fails to teach the use of aluminum chloride or chlorine as crystal modifiers for any purpose. It would require some form of hind sight to assert the prior art makes obvious the use of aluminum chloride or chlorine to increase the aspect ratio of the hemihydrate crystals, as claimed in claims 5-8.

## THE SPIRING PAPER

Claims 9 through 14 require the continuous of monitoring the aspect ratio, which is not suggested by the prior art, i.e. Baig and Jaunarajs et al. Baig teaches a continuous process, but doesn't address the aspect ratio of the calcium sulfate fibers. Jaunarajs et al teaches the production of soluble fibers of calcium sulfate anhydrite using a batch process. The Examiner cites the Spiring paper to teach continuously monitoring a process ability/capability. The Spiring paper does not relate to processes to calcine gypsum or to any other aspect of the claims in issue. The Spiring paper teaches a statistical approach to process control and doesn't teach anything pertinent to Claims 9 through 14. Clearly, the Spiring paper does not overcome the failure of Baig in view of Jaunarajs et al to suggest the use of alum to control the aspect ratio of the acicular calcium sulfate alpha hemihydrate crystals being produced as required by applicants' claims.

#### CONCLUSION

The prior art fails to suggest the use of aluminum chloride or chlorine to increase the aspect ratio of acicular calcium sulfate alpha hemihydrate crystals.

Accordingly, reconsideration of the rejection of claims 5 through 8 under 35 USC

USG-3399

§103 as being obvious based on US Patent 5,320,677 to Baig in view of US

Patent 3,835,219 to Jaunarajs et is requested.

As explained above, the prior art fails to suggest the use of alum to control

the aspect ratio of acicular calcium sulfate alpha hemihydrate crystals.

Accordingly, reconsideration of the rejection of claims 9 through 14 under 35

USC §103 as being obvious based on US Patent 5,320,677 to Baig in view of US

Patent 3,835,219 to Jaunarais et al and the Spiring paper is requested. It is

submitted that the claims are patentable over the prior art. Reconsideration of all

grounds of rejection is respectfully requested and an early notice of allowance is

solicited.

Respectfully submitted,

January 19, 2006

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